A new attempt to adopt a machine for SHS lining ceramics inside pipes

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Abstract:

In order to conduct centrifugal thermite research experiments in the laboratory, a special apparatus is required. A self-propagating high temperature synthesis machine with acceleration up to 350g was fabricated to accomplish experiments in laboratory settings. Then, thermite reaction of Ferro oxide III and Aluminium inside a pipe was performed to produce Alumina ceramic in the innermost layer and Ferro layer. Combustion synthesis is characterized by extreme heating rate, high temperature, and short reaction time. Centrifugal force facilitated the phase separation of multi-component products during the process. Preliminary tests were conducted prior to fabrication to realize reaction conditions.

Keywords: Ceramic-lined pipes; Centrifugal; Self-propagating high-temperature synthesis; SHS; Combustion synthesis; Thermite

1. Introduction

The self-propagating high-temperature synthesis (SHS) technique results in the in situ creation of composites from reactant substances through exothermic chemical reaction. The heat generated by the exothermic reaction to ignite and sustain a propagating combustion wave through the reactants creates the anticipated product (Fan et al. 2006; Graeve and Munir 2002; Ivleva et al. 2011; Kanakala et al. 2011; Odawara 1990; Odawara 2010; Pei et al. 2009; Salonitis et al. 2010; Wang and Yang 2007). SHS processes are characterized by high-temperatures; these characteristics make SHS an economical method for the industries compared to conventional ceramic processing. SHS is a feasible technique for preparing advanced ceramics, catalysts, and nanomaterials (Munoz et al. 1997; Patil et al. 1997; Sun et al. 2008).
Ceramic-lined composite pipe is a high-performance, heat-resistant, anti-corrosion and wear-resistant pipe that can be produced through the centrifugal SHS process which is spontaneous reaction propagation and rapid synthesis under the effect of centrifugal force. The reaction occurs in the inner pipe surface after melting the Aluminum in the first stage. The self-sustained exothermic chemical reaction propagates through a premixed powder in the form of a high temperature reaction wave with fast heating rates and short reaction times. Then it would be followed by phase separation and solidification of Al_2O_3 and Fe products. The product of centrifugal SHS reaction between Fe_2O_3 and Al is a metal-ceramic composite of Al_2O_3-Fe (Odawara et al. 1989; Rogachev and Baras 2007; Xu et al. 2010).
The adiabatic combustion temperature of the centrifugal SHS reaction can reach up to 2400ºC which is higher than the melting points of Al and Fe_2O_3 (Chatterjee et al. 2008; Patil et al. 1997; Sun et al. 2008). The advantage of this process is that once ignited, no further externally applied energy is necessary. Processing time is reduced, and the products are normally of high purity (Chatterjee et al. 2008; Merzhanov 1990; Yu et al. 2010).
The simple, rapid, and economical centrifugal SHS process was used to produce various types of metal-ceramic composite pipes. Ceramic-lined pipes are applied in several fields such as petrochemical, metallurgy, chemical and mining industries (Andreev et al. 2011; Lai et al. 1997; Munir et al. 2000; Zhou et al. 2010; Zhou et al. 2010).

Developing a test rig was necessary to conduct more research on centrifugal SHS products. In the current research work, a test rig was designed and fabricated to facilitate the study after performing a few preliminary experiments. The apparatus is as simple as possible to maintain the SHS process concept feasible and so it can be adopted in a wide range of exothermic ceramic lining techniques in laboratories. This apparatus facilitates research work in the field of centrifugal SHS process in industrial and educational fields.

2. Test Rig Fabrication

A centrifugal machine was designed to facilitate the SHS experiment utilized for laboratory-scale production of ceramic-lined steel pipes. Rotation speed, simplicity of use, ergonomic and safety factors, stability, and low vibration aspects were considered in machine development. The technical requirements for high-temperature and high-speed reactions in the small chamber were determined during preliminary tests.

2.1. Apparatus Design

Anthropometry information collected from 32 students in the 2007-2008 session at University of Malaya was adopted for the test rig design. It is expected to cover 95% of the population of potential users in terms of hand grip area and height (Schlick 2009). A cross-sectional view of the designed centrifugal machine is shown in Figure 1. The machine would have to undergo high centrifugal acceleration and rapid temperature rise during experiment. The steel pipe must be fixed inside the reaction chamber as shown in Figure 1.

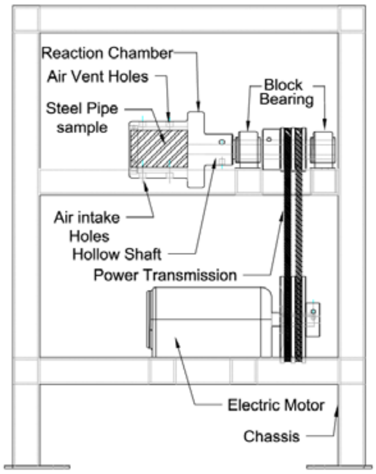

Figure 1: A cross-sectional view of the designed test rig

2.2. Fabrication

The reaction chamber is made of gray cast iron of internal and external diameters of 105mm and 85mm, respectively and lengths of 125mm. The reaction chamber is connected to a hollow shaft. There are six holes of certain angles considered in the chamber as shown in Figure 1, for ease of air flow inside the chamber to avoid pipe stagnation effect and deformation. The whole assembly is protected with guards and doors for safety. The reaction chamber, chassis, and power transmission are identified in Figure 2. The rotational power is transferred by two V-belts from the electric motor. A program embedded tunable inverter can control the reaction chamber's rotational speed in order to provide the required centrifugal acceleration which can reach from a=2g up to a=380g.

3. Experimental Procedure

The fabricated centrifugal machine was used for the production of ceramic coating in metallic pipes. Aluminum (< 75µm, 99% purity, Sigma Aldrich) and Fe_2O_3 (< 5µm, 97% purity, Sigma Aldrich) powders were dried for eight hours and mixed for 5 hours at a speed of 35rpm. The green powder stoichiometry mixture was kept based on equation (1):

$$Fe_2O_3 + 2Al \rightarrow Al_2O_3 + 2Fe + 836\text{ kJ} \quad (1)$$

A carbon steel pipe with a length of 110mm, inner and outer diameters of 69mm and 75mm, respectively, was used. The pipe was fixed inside the reaction chamber, while a refractory cap was attached to the pipe head. The pipe was charged with the green powder mixture while rotating at low speed. During the experiment, an embedded speedometer monitored the rotational speed of the pipe in the centrifugal SHS machine. Ignition of the charged powder mixture in the pipe was initiated by Oxyacetylene gas. The rotational speed was kept constant at centrifugal acceleration of a = 250g during reaction to ensure reaction uniformity and completion inside the pipe. The overall experimental setup scheme before and after reaction is shown in

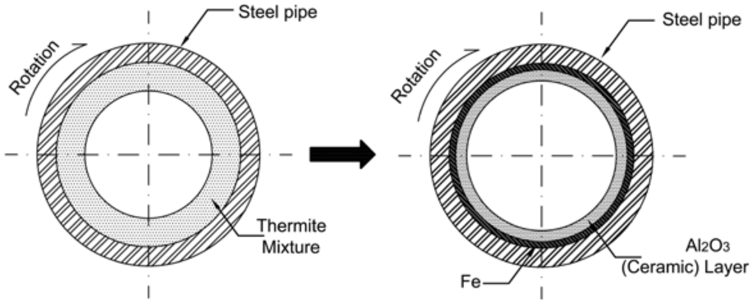

Figure 2: Schematic diagram illustrating steel pipe charged with a green mixture before reaction (left) and after reaction (right) while subject to centrifugal force

Normally, the SHS reaction is rather violent (Li et al. 2005), a sign that the reaction is beginning inside the pipe. In order to reduce reaction violence, Al_2O_3 was added to the mixture as inert diluent (Orru et al. 1996). The exothermic reaction between Fe_2O_3 and Al inside the pipe produced high temperature Al_2O_3 solid ceramic and molten Fe. Phase formation of products was followed by phase separation under the centrifugal force at high rotational speed (Orru et al. 1996; Yukhvid 1992). The pipe was kept rotating at the same rotational speed since the start of reaction until product solidification was complete. The cooled ceramic–lined composite pipe was taken out for characterization. Micro-hardness of the ceramic layer was measured at different points on the ceramic layer surface.

4. Results and Discussion

Structural formation occurred simultaneously with phase separation during the centrifugal SHS process. The phase formation and separation aspects of centrifugal SHS reaction between Fe_2O_3 and Al powders were studied. The microstructure of various layers was studied by scanning-electron microscopy (SEM). A typical ceramic-lined composite pipe produced by the fabricated machine is shown in Figure 3. The visual evaluation of the pipe revealed incomplete distribution in the innermost layer.Figure 3.a is the cross sectional view of the steel pipe. Figure 3.b demonstrates a typical magnified section of the formed layer, a macroscopic Fe layer and the pipe (steel substrate). Byproducts such as hot fumes and gases were produced during the reaction as well. In the preliminary experiments, due to high the temperature gradient, pipe deformations were observed due to the softening of pipe walls. There are six holes considered in the reaction chamber wall (Figure 1) in order to ease the airflow and reduce pipe deformation by disseminating the heat generated during reaction.

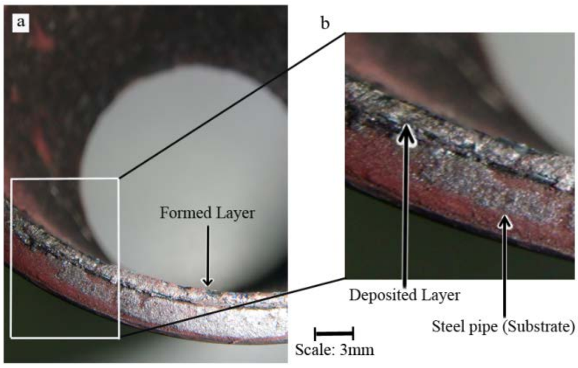

Figure 3: Photographic view of a typical product of centrifugal SHS reaction

A Bruker D8 Advance with a monochromatic Cu Kα source was employed for X-ray composition analysis of the ceramic layer surface. The X-ray diffraction pattern of the obtained products (Figure 4) elicits the formation of Al_2O_3 (Corundum), Fe and $AlFe_3$. In Figure 4, the most intensive peak of Al_2O_3 was detected at 2θ=37.69°.

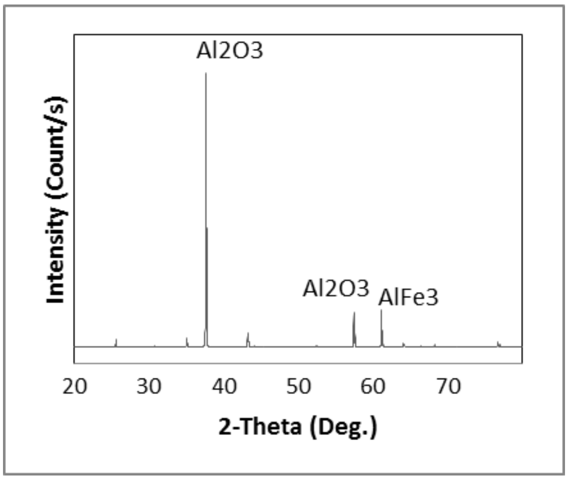

Figure 4: X-ray diffraction pattern of the centrifugal SHS products on inner surface

The product layers were separated according to their density and featured as a multilayered structure. The centrifugal SHS reaction led to the formation of a two-layered product inside the steel pipe. The SEM micrograph image of the product cross sectional view is in Figure 5. Phase separation of the two phases in bright and dark color is presented in the interface. The upper (lighter) layer constitutes corundum (Al_2O_3) and the lower (heavier) of the inter-metallic phase is $AlFe_3$ and pure Fe. The layer interface and particle diffusion from one side to another can be seen in Figure 5. The straight-line boundary between the phases of about 270μm shows the uniformity of layers thickness. Centrifugal acceleration was sufficient to form different phases with different densities. However, the dark phase penetrated into the bright phase in the central point of the boundary (Figure 5). Ceramic phase solidification occurred in the Fe rich phase. Either this is a sign of the solidified ceramic penetrating into a soft Fe-rich phase at high temperature, or the ceramic phase occupies a void generated due to out-gassing. This phenomenon improves the physical bonding of the two layers and can be considered micro-mechanical locking.

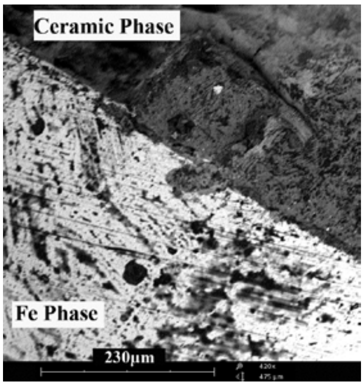

Figure 5: SEM micrograph of the boundary between ceramic and metallic layers

The hardness varies from point to point due to heterogeneity in phase-structure distribution in the inner surface of the ceramic layer. Hardness variation is directly related to the concentration of pores. The maximum hardness value recorded was 2365HV. Pipe curvature influences the compressive and tensile stresses generated in the layers (Odawara 1990). However, the bonding between layers and their mechanical characteristics present a successful process performed by the developed test rig.

5. Conclusion

The fabricated test rig seems feasible to be used in the laboratory in order to prepare ceramic-coated pipes using the centrifugal SHS method. Preliminary tests prior to fabrication improved the quality of the developed centrifugal SHS test rig. The apparatus can produce a wide range of SHS activating powders under different centrifugal accelerations and parameters. Phase formation was verified by XRD and SEM tests. Mechanically interlocking bonding was observed in different phases, which helped improve the bonding strength.

6. Acknowledgements

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